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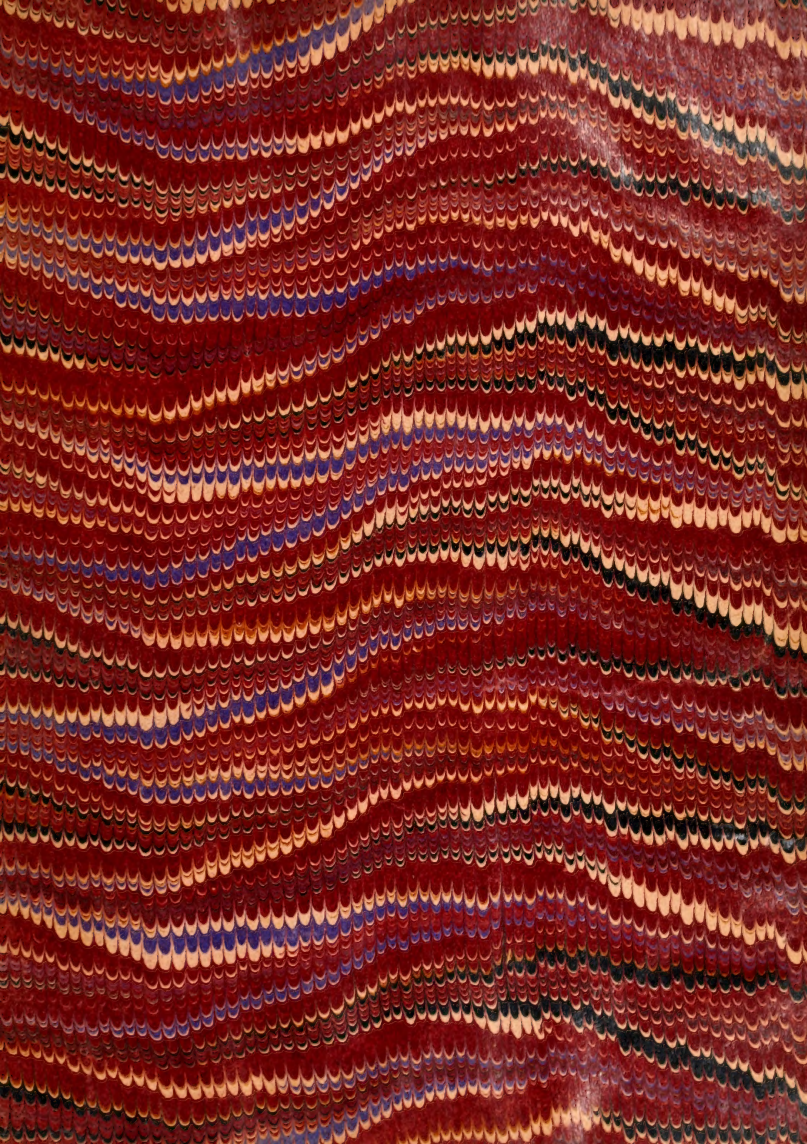
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An Investigation of the Constitution of the  
of the University of the Pacific (1902)

## Dissertation

Submitted to the Board of University  
Studies of The Johns Hopkins  
University in partial fulfillment  
of the Requirements for the  
Degree of Doctor of  
Philosophy

By  
The Johns Hopkins University

1902





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## Acknowledgments.

The author welcomes this opportunity to express his sincere thanks to President Paul Ramsey for the excellent instruction received in both lecture room <sup>and</sup> laboratory. This work was undertaken at his suggestion <sup>and</sup> carried out under his direction.

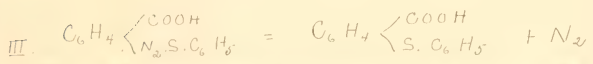
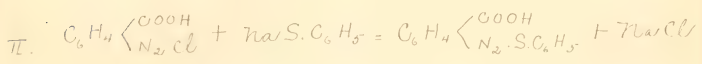
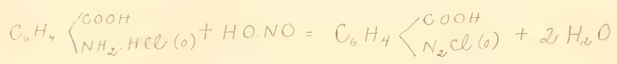
Thanks are also due Professor Mary Ferguson, Professor Black, Ed. Mackenzie and Stuart





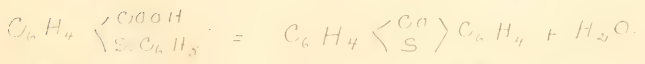
# Isolation

Phthalic acid is prepared from anthranilic acid by means of the following reactions:-



Phthalic acid is not isolated as an immediate diazo compound.

By the action of concentrated sulphuric acid upon phenylthioanthranilic acid we have obtained phthalic acid.



The next year Liebig<sup>(1)</sup> & Schultze<sup>(2)</sup> succeeded in isolating the diazo compound

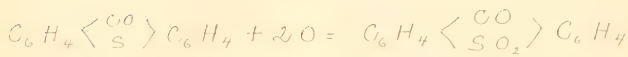
(1) Ber. d. chem. Ges. 33. 2471.

(2) Ann. chem. (Liebig) 263. 1.

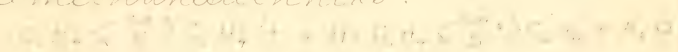


ground referred to, I also effected the following transformations, thus:

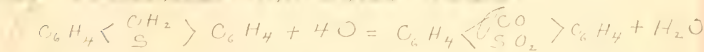
Thioanthrone can be oxidized by means of chromic acid or potassium dichromate to thionine.



Reduction of thioanthrone by hydrochloric acid or zinc dust gives diphenylmethanesulphide:



Oxidation of this last product gives at once to benzophenonesulphone, the intermediate compound which may be obtained in this way:

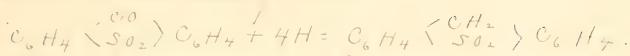


Reduction of benzophenonesulphone however gives one of the two possible intermediate compounds, viz. diphenyl





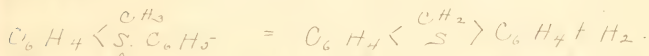
methanesulphonate:



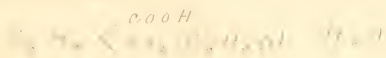
Further it was shown that orthodiazotoluenechloride could be transformed into orthotolylphenylsulphide:



and when the vapour of orthotolylphenylsulphide is passed through a heated tube of phosphorus, the following reaction occurs:



The compound of this reaction was subjected to phenylthioacetic acid to the action of dilute nitric acid & obtained a product to which they gave the formula



Analysis of this compound gave figures close to those required by the formula



Results of the acid were described:

(Rosen <sup>1</sup> & Cantel <sup>2</sup> in 1900 prepared an acid which appears to have the same structure as this oxidation product.

The reactions employed are as follows:

Orthochloranil, benzochloranil when treated with benzene & aluminium chloride yields orthochlorophenylbenzochloranil  $C_{12}H_6Cl_2$  & benzochlorophenylbenzochloranil  $C_{14}H_8Cl_2$ .

When this product is oxidized by means of potassium permanganate it passes to orthophenylsulfophenylbenzoyl acid  $C_{12}H_6SO_3$  &  $C_{14}H_8SO_3$  +  $3O = C_{12}H_6SO_3$  &  $C_{14}H_8SO_3$  +  $3O$ .

The properties of the two substances are quite different. The compound obtained by Dr. H. S. Schuster contained one molecule of water of crystallization & melted at  $100^\circ$ , when dehydrated at  $102^\circ$ . Crystallized from 95 per cent



alcohol it is deposited in fine needles.

Remsen & Canter's product crystallizes from alcohol in small plates grouped in clusters, is anhydrous, & melts at  $104^{\circ}$  ~~104.5~~.

The two compounds are clearly different, & since the plausibility of the structure assigned to each is so great, the investigation was undertaken with the view of studying the nature & extent of the differences more closely. In the first place an explanation of the discrepancy might thus be found.

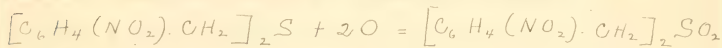
The probable explanation it appeared that Grabe & Schultze might have isolated this sulphoxide instead of the sulphone. But previous experience is known in which nitric acid is used



transformed into acetoxy sulphonic acid  
phenol, notably the following:-

Stenhouse<sup>1)</sup> showed that phenyl  
sulphide is changed into phenyl  
sulfone when heated for 1-2 hours with  
strong nitric acid.

Fabius<sup>2)</sup> succeeded in preparing ortho-  
nitrobenzylsulphone by heating ortho-  
nitrobenzylsulphide with fuming  
nitric acid in a sealed tube at 155°.



Ordinary concentrated nitric acid  
however, gave rise to the sulphonic acid.  
Potassium permanganate in ac-  
etic acid gave the sulphonic acid.

Potassium permanganate accom-  
plished the transformation to the  
sulphone in many cases where nitric  
acid produces only the sulphonic acid.

<sup>1)</sup> Ann. Chem. (Liebig) 140, 290.

<sup>2)</sup> Monats. f. Chem. 10, 381.





nitrosulphoxides, for example,

Märcker<sup>1)</sup> showed that benzyldithion is oxidized to benzyldisulphoxide by treatment with cold nitric acid (sp. gr. 1.3):



If heat is applied during the oxidation benzoic<sup>2)</sup> acid is formed as a result.

On the other hand, potassium permanganate converts benzyldithion into benzyldisulphoxide.<sup>(2)</sup>

Methylethylphenylamine,  $C_6H_5 \cdot CH_2 \cdot N(CH_3)(C_2H_5)$ , is transformed into dimethylethylphenylsulphoxide,  $C_6H_5 \cdot CH_2 \cdot N(CH_3)_2 \cdot SO$ , by the action of fuming nitric acid.

It is<sup>3)</sup> by means of potassium permanganate the sulphone,  $O_2S \cdot \langle C_6H_4 \rangle \cdot N \cdot CH_3$ , is obtained. The formation of nitrosulphoxides is also observed when ethylphenylamine,  $C_6H_5 \cdot CH_2 \cdot NH_2$ , and

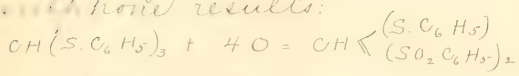
<sup>1)</sup> Ann. Chem. (Liebig), 136, 89.

<sup>2)</sup> Otto & Lüders. Ber. d. chem. Ges. 13, 1287.

<sup>3)</sup> G. S. G. Ber. d. chem. Ges. 13, 1287.



acetylthioacetophenone,  $\text{CH}_3\text{CO}(\text{S.C}_6\text{H}_5)_2$ ,  
 are treated with fuming nitric acid  
 (10% excess) and acetone of chloroform  
 and are treated with potassium  
 permanganate in acidic solution with  
 the following results:



If alkaline permanganate is used,  
 the tri-sulphone,  $\text{CH}(\text{SO}_2\text{C}_6\text{H}_5)_3$ , is obtained  
 when treated with nitric acid the  
 sulphido-sulphon is unidentified sub-  
 stance containing nitrogen.

Prof. B. B. Borison<sup>12</sup> stated that he  
 was studying the action of nitric  
 acid of various concentrations upon  
 organic sulphides but as yet no  
 paper by him has appeared upon  
 this subject.

<sup>12</sup> Jour. Chem. Soc. 1911, 1111  
<sup>13</sup> Jour. Chem. Soc. 1912, 1111  
<sup>14</sup> Jour. Chem. Soc. 1913, 1111



## II. Preparation of Material.

Thiophenol,  $C_6H_5.SH$

The thiophenol used in this investigation was prepared by the following method, which is a slight modification of that one proposed by Otto.

To 150 cubic centimetres of water, which is heated to boiling in a round bottom flask attached to a retort condenser, 80 grams of zinc dust is added <sup>at</sup> without further heating. Hydrogen sulphide is then added in 100 grams all allowed to flow in in small portions. The reaction is accompanied by a hissing sound. It is allowed to subside before the next portion of the chloral is added. The flask is shaken continuously. After all of the chloral has been added the flask is heated for about twenty minutes.



water. The rate of the reaction  
increases considerably during the addition  
of the latter portions of the chloride, it  
is well to apply heat then also.

A mixture of 450 cubic centimetres  
of concentrated hydrochloric acid <sup>and</sup>  
675 cubic centimetres of water is placed  
in a six-litre round flask. Cool well  
by immersion of this flask in ice-water  
Add a few drops of a solution of cop-  
per sulphate. Add 25 grams granulated  
zinc. Continue to cool the solution. It  
now gradually forms small portions the  
white material obtained in the reac-  
tion described above. This should also  
be cooled. Introduction of crushed ice  
into this flask during the reduction  
is also advantageous.

When the evolution of hydrogen





becomes moderate, add 100 grams <sup>and</sup> allow to stand (with cooling) for a half hour or more. Add more 20 grams more of zinc, dust <sup>and</sup> heat on the water bath until the zinc dust sinks to the bottom of the flask, showing that the solution is no longer acid. The acid column condensed should be attached to the flask during the heating. Cool the contents of the flask, & add a large excess of concentrated hydrochloric acid. Distill in steam.

The flask should be heated gradually, whenever a very violent evolution of hydrogen takes place suddenly. Should this occur it may be readily checked by pouring cold water over the flask. The whole process over <sup>and</sup> as the first portions are especially rich in

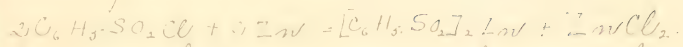


best to use two receivers. In the first  
the kerosene collects at the bottom and  
oil which can be separated from the  
water by means of a separatory funnel.  
The remaining liquid should be extracted  
with ether. Most of the ether is evaporated  
off. The residue is then dried by  
means of fused potassium carbonate.  
I distilled "Primer stone" which should  
be used in these distillations  
as when obtained, the boiling point  
is  $168^{\circ}$  uncol. Pure kerosene accord-  
ing to Stenhouse boils at  $172.5^{\circ}$ . The  
yield is low as the products are  
poor. One kilogram of benzene  
gives about 500 grams  
kerosene. Not pure, though.

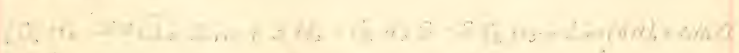
The reactions involved are:- First,  
the reaction to the point of the



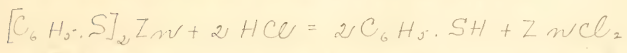
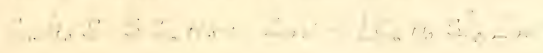
benzenesulphonic acid:



Second, the reduction of this to phenyl disulphide:



Third, when the result here is heated with zinc dust the product acidified, chiorphenol is obtained.



About two mols are required for the preparation of 600-800 grams of this acid by the above method.

For the preparation of chlorophenol in small quantities <sup>very</sup> in a short time the method proposed by Stadler<sup>(1)</sup> may be used to advantage.

When pure, <sup>very</sup> in concentrated form chlorophenol does not possess a disagreeable

<sup>(1)</sup> Ber. d. Chemiker 12, 2575



and when it is used as a dressing  
for a large burn of course however it is  
exceedingly unpleasant. Fresh blood is  
which adheres to it, highly effec-  
tual in curing ulcers and other  
wounds which are difficult to  
heal. Its vapor attacks the eyes <sup>and</sup>  
mucous membrane of the nose <sup>and</sup>  
throat with very unpleasant results.

### Chloroformic Acid

*Chloroformic Acid*

A mixture of 40 grams chloroform  
60 grams sodium hydroxide <sup>and</sup> 300 cubic  
centimetres water is warmed to 60°  
the chloroformic acid is added  
as a solid form as dropping funnel  
a thin stream. For the diazotization  
40 grams anthranilic acid, 20 grams





centimetres water are used, the temperature being kept between  $2^{\circ}$  &  $5^{\circ}$ .

When the sodium chlorhenolate is mixed with chlorosulphonic acid the alkaline solution is coloured red, & an evolution of nitrogen takes place. The mixture is placed in the flask & immersed in the water-bath for an hour, & until the colour has changed to yellow. The solution is acidified by phenyl chlorosulphonic acid & the mixture is allowed to stand until the colour has changed to yellow. The mixture is then cooled slightly. The cooled solution is filtered from the chlorosulphonic acid which is always formed by oxidation of the phenol. The residue



of phenyl disulphides can be used in  
transforming thiophenol by simply heating  
with zinc dust & acidifying the filtrate  
again and fusing carefully. The  
thiophenol disulphide acid is more  
soluble in water, soluble more than is  
expected after it has become perfectly  
free, when washed with water.  
I found on a porous plate

From 75 grams of thiophenol and  
about 6.5 or 70 grams of the crude pro-  
duct are obtained; the theoretical yield  
is 74 grams. This product is, however,  
quite impure, <sup>yes</sup>, after complete puri-  
fication, the quantity obtained is about  
25 grams.

I wished to purify the product it  
is found best to treat as follows:

Distill in hot alcohol until the



solution is quite concentrated. Allow to  
settle partially, and the mass of fine  
crystals which separates converts the so-  
lution almost into a solid. Filter off  
the crystals, using a 16 in. plate or  
Buchner funnel. Separating the dark,  
colored mother liquor and completely re-  
move the fresh separate twice with cold  
water. It will be found that but a few  
crystallizations are necessary to bring the  
mass to a state of comparative purity of  
the mother liquor is a heavy, white,  
thick crystals with much substance.

In the last crystallization it is best  
to use alcohol of about 80% and the  
residue contained in the mother liquor  
may to a certain extent be recovered,  
but a quantity of black, tarry material  
is always present. Phenylthiocarbonyl



acidic cellular carbonic acids, melts at 100°  
and when heated strongly, subliming  
easily without decomposition. Long paper  
test. In leaflets. If heated to about 100°, it  
softens, and being heated to 100° or 110°  
it changes to a white, cottony mass, which is  
found in beautiful, cottony masses, with  
a faint, soft, blue color. The color is  
then affected by warming with  
concentrated sulphuric acid, in which  
it dissolves with a very deep, green  
fluorescence.

The methylacetic acid is a colorless  
liquid, soluble in water. It is almost  
insoluble in cold water. It is soluble  
in benzene, methyl ethyl & isopropyl  
alcohol, ether. It is fairly in pure  
water and

Can be distilled, and is a colorless



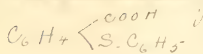


Potassium ammonium <sup>the</sup> soluble salts  
<sup>the</sup> are collected. The barium salt is  
 quite soluble but apparently is incapable  
 of being crystallized.

Analysis of the acid gave the fol-  
 lowing results:

1.0000 g. acid + 1.0000 g.  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$   
 gave 1.0000 g.  $\text{BaSO}_4$

Calculated for



Found.

C 67.78

67.40

H 4.39

4.45

S 13.93

14.19



is a solution of 1.5 g. of the acid in 10 ml. of the  
calcic glycolic acid.

When treated with dilute nitric acid  
several products result, the principal  
one being phenylacetic acid or carbonic  
acid. The compound described by  
Graef & Schultze is also formed, but  
this is not a product when a  
nitric acid is used. There is also formed  
in smaller amount a product melting  
at 200°-220° which is not a  
solid.

This product will be taken up in  
water.

10. Phenylacetic acid or carbonic acid  
$$C_6H_5 \begin{matrix} \nearrow COOH \\ \searrow SO_2 \end{matrix} C_6H_5 (O)$$

One gram of phenylacetic acid  
was dissolved in 10 ml. of water and  
with 10 ml. of concentrated hydrochloric



acid [1 vol. concentrated nitric acid to  
2 vol. water], <sup>45</sup>g. the mixture boiled.  
The material becomes dark <sup>45</sup>g. the  
brown fumes are given off. The  
substance which at first floats on the  
brown concentrate sinks to the bottom.  
The heating is stopped, water is added  
the material allowed to stand until  
it has solidified. It is then put into  
water. It is washed & treated with dilute  
hydrochloric acid with small quantities of  
hydrogen peroxide. Some reddish granules, leav-  
ing a yellow mass behind which is  
then crystallized from water & alcohol.  
It can also be crystallized from water  
or alcohol & alcohol it is very  
soluble. The form in which it is precipitated  
is large rhombohedrons which show  
a tendency to form parallel growths.



From methyl alcohol the crystals are  
more elongated <sup>and</sup> assume the prismatic  
form that is first obtained when crystals  
are yellow <sup>and</sup> melt at  $161^{\circ} - 162^{\circ}$ . By  
repeated crystallization however all  
obtained colorless <sup>and</sup> transparent melt-  
ing at  $161^{\circ}$ . By crystallization from  
ethyl alcohol these crystals  
are not very white needles arranged  
in bunches which resemble very much  
the product described by Beilstein &  
Schultes; <sup>and</sup> in fact, as there two  
may be present in the solution it is  
very difficult in many cases to say  
whether the product is homogeneous  
or not. However, if the crystals  
are crystallized from  
ethyl alcohol these needles are ob-  
tained, which consist of phenyl-





phosphotetracarbonic acid containing one molecule of water of crystallization. When crystallized from water alone, the same is observed, but another phenomenon is noted, viz: - the tendency on the part of the hydrous form to 'slake' back to the anhydrous form when in contact with water.

The hydrous form has not a definite melting point. It begins to soften at about  $80^{\circ}$ - $85^{\circ}$ , losing water of crystallization at  $100^{\circ}$ - $105^{\circ}$ , <sup>then</sup> melting completely at  $164^{\circ}$ . If heated suddenly to a temperature of  $180^{\circ}$ - $200^{\circ}$  it melts with foaming.

On crystallization of the hydrous (needle) form of the acid from methyl or ethyl alcohol quiescence



anhydrous rhombohedral form.

Phenylsulphide monocarbonic acid, as has been mentioned, is quite soluble in ordinary alcohol, somewhat less so in methyl alcohol. It is very soluble in propyl alcohol, readily soluble in chloroform, <sup>and</sup> difficultly in ether. Hot water dissolves it with great difficulty, <sup>and</sup> in cold water it is practically insoluble. It is unchanged by heating with concentrated or fuming nitric acid. Heated with concentrated sulphuric acid it dissolves, forming a red liquid with a brown, green fluorescence. With the hope of obtaining a compound soluble in chloroform this solution was heated for several hours at  $110^{\circ}$ , <sup>and</sup> after cooling poured into water.



The dark colored material which separated did not, however, seem capable of crystallization.

Summation of the analyses and the following results were obtained:  
 0.1812 gram gave 0.0647 gram  $H_2O$  & 0.4217 gram  $CO_2$ .

II 0.2035 " " 0.0711 " " 0.4693 "  $CO_2$ .

III 0.1620 " " 0.1581 "  $BaSO_4$ .

IV 0.1615 " " 0.1575 "  $BaSO_4$ .

Calculated for

$C_{10}H_{10}O_4$

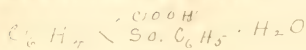
		Found	
		I.	II.
C	63.38	63.46	62.89
H	4.10	3.99	3.91
S	13.12	13.40	13.36

As it is impossible to obtain the hydrous acid free from the anhydrous an accurate determination of the  $H_2O$  of crystallization could not be made.



The butyric acid obtained from the following -  
0.025 gramme dried acid butyric anhydride  
at  $105^{\circ}$  0.0133 gramme.

Calculated for



Found

12.0

6.82

6.88

Salts of *Thymoliphenylacetic acid*.  
(Acid)

These prepared and well crystallized  
exhibit the phenomenon of super-  
saturation.

Barium Salt. This salt was pre-  
pared in the usual manner by digest-  
ing a barium salt with water & an excess of  
barium carbonate, filtering and evaporat-  
ing to crystallization. White needles  
in prisms are deposited, which are





Stems arranged in bunches

Upon analysis the following results were obtained: -

0.1538 gram of anhydrous salt lost at  $110^{\circ}$  0.0229 gram  $H_2O$ .

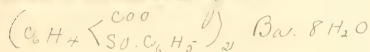
0.2436 " " " " " " " 0.0447 "  $H_2O$ .

0.14645 " of salt dried at  $110^{\circ}$  gave 0.0533 "  $BaSO_4$ .

0.1690 " " " " " " " 0.0623 "  $BaSO_4$ .

0.1758 " " " " " " " 0.0648 "  $BaSO_4$ .

Calculated for



Found

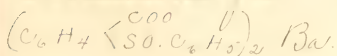
$H_2O$

8.25

8.45

8.47

Calculated for



Found

I. II. III.

Ba 21.89

21.42

21.69

21.62

Strontium Salt. - This was prepared

from the same acid as the barium salt. It is apparently identical with the barium salt. It is soluble



it being. The crystallized salt is efflorescent, so an accurate determination of the water of crystallization could not be made.

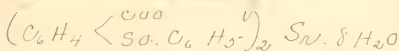
0.2500 gram of crystallized salt heated at 110° gave

0.2516 " " " " " " " 0.0491 "

0.2025 gram of salt dried at 110° gave 0.0636 gram  $\text{SrSO}_4$ .

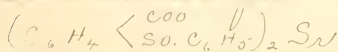
0.1869 " " " " " " " 0.0583

Calculated for



$\text{H}_2\text{O}$  19.95

Calculated for



$\text{H}_2\text{O}$  19.95

Found

I. II.

19.46 19.51

Calcium Salt:— This salt was prepared in the same manner as that used in the strontium & barium salts. It is apparently isomorphous with these crystallizing in prisms.

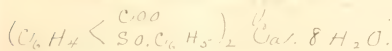


analysis - calcium

1.2774 gram of crystallized salt lost at  $110^{\circ}$  0.558 gram  $H_2O$ .

.720 gram of salt dissolved in 10 cc water, 2.502 gram dry.

Calculated for

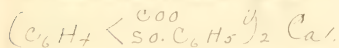


Found:

$H_2O$  21.36

20.53

Calculated for



Found:

Ca 7.50

7.37



8. Comparisons of the compound described by Wm. Schultze, to which they correspond the formula,  $C_{10}H_8O_2$ ,  $C_{10}H_8O_2$ ,  $C_{10}H_8O_2$ .

Wm. Schultze prepared the product by the action of nitric acid and sulfuric acid on phthalic anhydride. Repeating the experiment described by them, & following as closely as possible the instructions given, I have found that in most cases the compound above described as the end product resulted in large quantity, but frequently very little of the desired product was obtained. By the use of concentrated or fuming nitric acid the product is almost wholly the one described by Wm. Schultze.

To 10 grams of phthalic anhydride acid, contained in a large test tube,





about 10 cubic centimetres of fuming nitric acid were added slowly. Violent action took place immediately. A black, red solution was obtained. If ordinary concentrated nitric acid is used it is necessary to heat in a water bath for a few minutes to effect complete solution. The product is poured into water & the oil which separates allows itself to be separated & hastened by rubbing with a little cold water. The yellowish mass should then be pulverized, washed with water & treated with small quantities of ether on the filter in order to remove the red-stained impurities. The product remains almost perfectly white. It is soluble in warm alcohol, in which it is very soluble & the solution obtained is having



red color. After several centimetres of water are added, the solution begins to crystallize.

These white needles arranged in small bunches, if not too dense the liquid will become almost solid. These crystals melt at  $92^{\circ}$ , when dehydrated at  $105^{\circ}$ , melt again at  $152^{\circ}$ . From 10 grams phenylthiosalicylic acid 7.5 grams of the product are obtained.

The compound is very soluble in water, and in alcohol as before. It appears to be perfectly uniform. But after the third or fourth crystallization a decided change is observed: in addition to the needles, a white, fine-grained substance is formed. The mixture does not show any













located at the surface of the compound  
 substance is deposited in a solid  
 fine white needles arranged in large  
 bundles. The compound is soluble in  
 water and alcohol. It melts  
 at  $180^{\circ} - 184^{\circ}$ .

Evidently the compound is a  
 hydrate of the compound.

A series of experiments were made  
 to determine the following results:

0.1440 gram lost at $110^{\circ}$	0.0073 gram $H_2O$	= 6.46
0.1716 " " " "	0.0108 " " "	= 6.27
0.1549 " " " "	0.0099 " " "	= 6.39
0.1486 " " " "	0.0094 " " "	= 6.23







Percentage of

	C.	H.	N.
1	56.54	56.34	56.35
2	3.71	3.67	3.87

Substance dried at 135°.

Substance dried at 135°.

$H_2O$  0.2454 gram

Percentage

C. 56.54

H. 3.67

The compound also contains a  
small amount of water of crystallization.

When the substance is heated  
it melts at 135° and loses water of crystallization.

The following results were obtained from analysis of the substance.

0.2454 gram  $H_2O$  0.2454 gram  $CO_2$ .

Percentage

C. 48.85

H. 3.67



The acid melting at  $211^{\circ}$ - $214.5^{\circ}$ , a low m.p.  
for a solid acid. It is not soluble in water,  
is difficultly soluble in alcohol, and soluble  
in ether. An ethyl ether solution is not  
very stable. When dried it becomes  
solid by evaporation. It is not soluble in  
benzene, but it is soluble in chloroform.  
In some cases it has been found to  
be soluble in benzene, but this is not  
characteristic. In the dry state it is  
a solid, but it is easily converted into a  
liquid by the addition of a small amount  
of water. It is not soluble in water, but  
it is soluble in alcohol, and it is soluble  
in ether. The latter may be due  
to the presence of a small amount of  
water, which is not very important.





Experiment 1. The mixture was heated  
for 10 minutes.

1.0000 gram of salt was found to give the following result.

0.1252 gram salt dried at 130° gave 0.0400 gram  $BaSO_4$ .

Percentage of  
 $Ba$  = 32.00  
" " " " = 68.00

Theoretical salt

0.1252 gram salt dried at 130° gave  
- at 130°.

0.1252 gram salt dried at 130° gave 0.0400  
gram  $BaSO_4$ .

Percentage of  
 $Ba$  = 32.00  
" " " " = 68.00

These figures are not far from those  
calculated by other means.



action of ortho-allylphenolic acids, but no evidence has been secured pointing to such a connection. It is suggested only as a possibility.

In the oxidation of allylphenols, allyl groups by conversion of  $\text{CH}_2$  to  $\text{CO}_2$  are converted to a carboxylic group, and small quantities of formaldehyde are also formed. It is difficult to obtain a definite result. The allyl group is found as an oxidized carbon and carbon dioxide and carbon monoxide are also formed.

Formaldehyde is not present in the oxidation of allyl phenols, but it is present in the oxidation of allyl alcohol.

The allyl group is found to be present in a group of allyl phenols. The allyl group is found in the oxidation of allyl phenols, but it is not present in the oxidation of allyl alcohol.



forming a yellow solution. When  
standing, long fine sticky yellow needles  
arranged in bunches are deposited. If  
these are dried & exposed to the air for  
some time they do not dissolve in  
water although upon the addition of  
a small quantity of ammonia they  
dissolve they decompose readily. The solu-  
tion when concentrated gives a glassy  
brittle precipitate which when long  
left standing, cracks & finally crum-  
bles & is covered with a white powder which  
is slightly soluble in water.

The substance has no reaction  
to litmus and alcohol solution. It  
dissolves readily in carbon disulfide, chloroform,  
benzene & is sparingly soluble in ether.  
The addition of ammonia



It then treated with concentrated sul-  
phuric acid, becoming yellow & red, as  
an oblique white, brown, brown, &  
yellow, but as a whole, at a higher  
temperature, however, it is exactly  
the same, leaving a yellow residue.  
If heated it heated again, the color  
changes to brown, whether it is  
upon heating into water a yellow so-  
lution is obtained. It is very slight  
soluble in water, but a yellow color  
the solution becomes etc.





III. Oxidation of Phenyl-hydrocyclic  
Acid by means of Potassium

By the use of potassium permanganate it was hoped that the acid could be obtained by Arsenic & Carter would be formed. It was to say, however, the product is not identical with cheirulonic, although its general reaction & analyses seem to show that it should. It is represented by the structure  $C_6H_4 \begin{matrix} \nearrow COOH \\ \searrow SO_2 \cdot C_6H_5(O) \end{matrix}$

Three facts will be discussed later.

IV. Phenyl-hydrocyclic Acid  
 $C_6H_4 \begin{matrix} \nearrow COOH \\ \searrow SO_2 \cdot C_6H_5(O) \end{matrix}$

For the preparation of this acid, the phenyl-hydrocyclic acid was prepared by the method of Arsenic & Carter, and was found to be identical with the product obtained by the method of Arsenic & Carter.



Carefully examined & tried to determine

Low pieces of the bright, sparkling  
acid were placed in a few bottles  
first containing about two litres of  
water. The flask was heated and water  
boiled in the water on one side of the  
flask. The ground potassium permanganate  
was added from time to time in  
small portions until slightly more  
than the calculated amount has  
been used. The oxidation proceeds  
rapidly. The reaction is complete  
in about one hour & a half. The  
amount of potassium permanganate is known  
by means of a few pieces of alcohol.  
After filtration the solution is evaporated  
to 10, 50 or 200 cubic centimetres.  
After cooling it is decanted into the



hydrochloric acid when phenylsulphonic  
anhydride is precipitated as  
an oil. Solidification is induced by  
cooling with ice or brine. It solidifies  
for white masses when purified. It  
is insoluble in water but is best crys-  
tallized from benzene, when it is de-  
posited in the form of white needles  
melting at  $143^{\circ}$ . Other solvents than ben-  
zene are unsatisfactory for use in crystal-  
lizing this acid. It is extremely soluble  
in alcohol, ether, and light petroleum. It  
is also soluble in all acetone and is  
almost insoluble in water. It is  
soluble in benzene and can be crystallized  
from alcohol or acetic acid with  
addition of water. But in most cases  
it appears as an oil. When deposited  
from a solution in ether or light petroleum it



in clusters of needles, which when  
heated quickly between glass plates, <sup>the</sup>  
the melting point taken immediately  
does not melt at  $143^{\circ}$ , but often much lower  
- usually near  $100^{\circ}$ . If allowed to stand  
in contact with the air for a short  
time, however, the highest melting-  
point is obtained. This seems to indicate  
that a state of crystallization is  
attained, but as but one compound  
is obtained when distilled water is added  
alone, some of the acid dissolves <sup>the</sup>  
upon cooling, along with the syrup.  
These cannot crystals are obtained. If  
a mixture of water and alcohol is added  
the crystals are more numerous and  
sharper. Beautiful clear prisms  
are frequently obtained which  
seem to be of the trichlinic





of rhombohedral hexagonal crystals.  
They contain 12 molecules of water  
and 10% of loss on heat. These crystals melt  
at  $57.5^{\circ}$ .

The reactions of the hexagonal rhombo-  
hedral crystals are as follows: They are in-  
soluble substances. Apparently no decomposi-  
tion takes place when heated to  $170^{\circ}$ .  
Above this some volatile products are  
formed, but high heat is necessary to  
bring about marked changes in color. It  
is attacked by dilute or concentrated  
nitric acid.

The crystals are soluble in water and  
alcohol, soluble with caustic potash at  
 $100^{\circ}$  to  $120^{\circ}$  and in strong acids. It is  
insoluble in concentrated sulfuric acid.  
It is soluble in strong mineral acids and  
in strong organic acids.



mass which states the whole is dissolved  
Upon acidifying the mass of hands,  
noticed, the solution became  
on all volume & recrystallizing from  
alcohol the fine white crystals which  
were deposited, white needles of  
the cold reaction with ferric chloride  
made with that given by a speci-  
men of salicylic acid. I found  
that the product is really salicylic  
acid. These products are what we  
should expect from a substance hav-  
ing the formula given above.

Properties of phenylphenanthrocar-



Some acid when boiled with aqueous potassium hydroxide for six hours, the same result when heated in two hours in boiling pyridine, in which substance it is very soluble.

Heated with concentrated sulphuric acid at 150°C. for 2 hours, a beautiful blue coloration is obtained. The color disappears on dilution.

The alcoholic solution is colorless and does not solidify on cooling to -78°C.

The compound is very hygroscopic. It is soluble in benzene, ether, and chloroform. It is insoluble in carbon tetrachloride, carbon disulfide, and carbon monoxide. It is soluble in alcohol. The melting point is 207°-208° under 1 mm. Hg pressure.



are also observed when the salts of the  
 acids are examined:

Analysis of the rhombohedral crystals  
 obtained from a saturated solution of the  
 anhydrous acid gave the following results:  
 0.2002 gram acid melting at  $51.5^{\circ}$  lost at  $100^{\circ}$

0.0183 gram  $H_2O$ .

0.1828 gram acid melting at  $51.5^{\circ}$  lost at  $100^{\circ}$

0.0179 gram  $H_2O$ .

Calculation for

$C_{10}O_4H_2$

Found

Found

I.

II.

$H_2O$  9.35

9.14

9.79

0.1986 gram of anhydrous acid gave 0.0656 gram  $H_2O$   
 0.4329 gram  $CO_2$ .

0.2177 gram of anhydrous acid gave  
 0.0699 gram  $H_2O$  0.4609 gram  $CO_2$

0.2177 gram of anhydrous acid gave 0.0699 gram  
 0.4609 gram  $CO_2$





0.127 gram of anhydrous acid gave 0.389 g. of

Calculated for



Found.

E. K.

C	59.51	59.44	58.86
H	3.85	3.70	3.77
S	12.23	12.07	12.14

0.127 g. of anhydrous acid gave 0.389 g. of

Calculated for  $C_6H_4(SO_2C_6H_5)_2$  salt as crystallized slowly from solution at 100°C. beautifully white, in small clusters, containing nine molecules of anhydrous acid. The crystals were not uniformly nine molecules in number.



The salt loses nearly all its water of  
 crystallization at  $115^{\circ}$ , but does not give  
 up the last portion - apparently, half  
 a molecule - until  $175^{\circ}$  has been reached.  
 This at first caused an error in the  
 analysis of the salt, the percentage  
 of water being found to be less than  
 the proportion of the salt. After  
 studying more carefully, when it was  
 found that the last traces of water  
 were not removed under  $175^{\circ}$ .

0.2156 gram of crystallized salt lost in  
 weight at  $115^{\circ}$  0.0377 gram  
 0.1779 gram of anhydrous salt lost in  
 weight at  $115^{\circ}$  0.0420 gram, at  $175^{\circ}$  0.0429 gram  
 0.1677 gram dry salt gave 0.0407 gram  $H_2O$  at  $184^{\circ}$   
 0.1512 " " " " 0.0363 " "



Calculation for

$(C_6H_4 \langle \overset{COO}{\underset{  }{SO_2}} \cdot C_6H_5 \rangle)_2$ Cal. $9 H_2O$		Found.	
		<u>I.</u>	<u>II.</u>
$8 H_2O$	21.77	21.75	21.63
$9 H_2O$	22.37	22.21	22.10

Calculation for

$(C_6H_4 \langle \overset{COO}{\underset{  }{SO_2}} \cdot C_6H_5 \rangle)_2$ Cal.		Found.	
		<u>I.</u>	<u>II.</u>
Cal. 7.11		7.14	7.06

When the salt is heated, it loses all water of hydration, as judged by analysis, and is left possibly not uniform, consisting apparently of hydrous & anhydrous parts. Determinations of the amount of water of crystallization have been made, and show that the material is not completely anhydrous, and that the heat has been reached. This is probably similar to the case of the other salts when heated.



Weight of dried salt 0.0040 gram.  
Weight of crystallized salt lost in weight at 120°  
0.0040 gram.

Weight of dried salt 0.0058 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0058 gram.

Weight of dried salt 0.0076 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0076 gram.

Weight of dried salt 0.0098 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0098 gram.

Weight of dried salt 0.0068 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0068 gram.

Weight of dried salt 0.0781 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0781 gram.

Weight of dried salt 0.0681 gram.  
Weight of crystallized salt lost in weight at 175°  
0.0681 gram.

Calculated for

$(C_6H_4 \langle SO_2 \cdot C_6H_5 \rangle)_2 Ba \cdot 1\frac{1}{2} H_2O$  Found.

	I	II	III	IV
H <sub>2</sub> O	2.66	3.28	—	—
SO <sub>2</sub>	3.93	3.30	3.31	3.40





Calculated for  
 $(\text{C}_6\text{H}_5)_2\text{C}(\text{SO}_2\text{C}_6\text{H}_5)_2 + 13\text{H}_2\text{O}$

Strontium Salt: - As in the case of Barium salt a crystallization sufficient to furnish an analytical sample is very difficult to obtain, and consequently the molecular weight of the salt is not reliable. The salt is not completely anhydrous, and the determinations of the amount of water retained below that temperature could be made.

0.298 gram crystal salt below 100°C.  
0.800 gram dehydrated salt gave 0.0085



Calculated for  
 $C_6H_4 \langle \overset{C_{100}}{SO_2} C_6H_5 \rangle_2$  Sr. 72.11 H<sub>2</sub>O

72 H<sub>2</sub>O 8.14

Found.

18.18.

Calculated for

$C_6H_4 \langle \overset{C_{60}}{SO_2} C_6H_5 \rangle_2$  Sr.

Sr 14.35

Found.

14.20.

It is very soluble in benzene.

$C_{10}H_8 \times C_6H_5 \times C_6H_5$

It is soluble in benzene and is decomposed by  
 sodium metal. It is also decomposed by  
 sodium metal, forming sulphuric acid. Cracks  
 Schultze<sup>(2)</sup> also obtained it by oxidation  
 of a hydrocarbon of molecular weight 100.



It is found that a compound of molecular weight 100 is  
 identical with the one obtained by  
 Schultze. It is also identical with the one  
 obtained by Schultze.

It is found that a compound of molecular weight 100 is  
 identical with the one obtained by Schultze.



the action of concentrated sulphuric acid  
in order, if possible, to effect a dehydro-  
genation similar to the one which is accom-  
plished in the case of aliphatic thiocarbonyl  
compounds. The compound, however, did not  
decompose, and the reaction was not  
successful. The compound was found to be  
stable, and no reaction was observed with  
water; a caseous precipitate was  
formed, which, after being filtered off,  
was found to be a solid, and was found to  
be a crystalline, very long chain  
compound, which was deposited in a very  
slightly yellowish form, and which melted  
at 100°C.

The color reactions which are given  
by the substance for identification









a. deep green fluorescence.

Analysis gave the following results:  
0.1294 gram gave 0.0401 gram  $H_2O$  & 0.3023

Calculated for  
 $C_6H_4 \begin{matrix} SO_2 \\ \backslash \end{matrix} C_6H_4$

Found.

C 62.00

62.00

H 3.30

3.30

Calculation of the following compounds  
is given below.



The compound was prepared by  
heating the substance with sodium  
hydroxide in a glass tube. The  
substance was heated to a  
red heat at a temperature  
of about 300°C. The water was  
driven off and the residue was  
the sodium compound. The



while becoming liquid <sup>the</sup> assuming a  
bright red color. The mass was then  
heated in a sulphuric acid bath to  
150° for several hours for the purpose  
of expelling phosphoric anhydride.

After cooling, it is treated with water  
and filtered. The material is then dried be-  
tween sheets of bibulous paper. The color  
is then a pale yellowish brown.  
It is then perfectly free from water  
and volatile.

The product is a solid mass  
the consistency of a resin. It is  
very soluble in water and alcohol  
at 100° but not in ether. It is very  
soluble in benzene, very difficultly soluble in  
gum. In contact with the air the  
material is very stable and does not



is decomposed by the moisture, as  
the substance is very hygroscopic.

On heating it with water, the  
phenylanthrone is decomposed  
and is precipitated.

During the treatment of this acid  
with concentrated sulfuric acid, the  
temperature of the sulfuric acid is

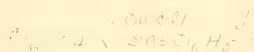
raised to  $100^{\circ}$  in the case of the  
phenylanthrone. The chloride is formed. The chloride  
is best crystallized from acetone. When  
this is heated it decomposes in small pieces  
which melt at  $121^{\circ}-123^{\circ}$ . It is  
very soluble in water, in the  $90\%$  alcohol,  
in ether, in benzene, acetone, and  
chloroform.

When the phenylanthrone is heated  
with sulfuric acid, it is decomposed  
and the phenylanthrone is precipitated  
as a solid substance.



It determined in a few minutes the  
soluble melting at  $75^{\circ}$  to  $80^{\circ}$  was made  
by boiling with aqueous potash.  
A few additions with dilute acids  
treating with silver nitrate.

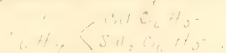
Calculated for







Ortho-oxalyl-dichloride,  $\text{C}_2\text{O}_2\text{Cl}_2$ .



This compound was first prepared by  
Liebig & Wöhler by heating oxalyl chloride  
with benzene & aluminium chloride. The  
compound is a colorless liquid, boiling at  
100°C. It is soluble in benzene & ether.  
It is a powerful oxidizing agent, and  
is used in the synthesis of many organic  
compounds. It is also used in the synthesis  
of aluminium chloride.

Both the acid & the chloride are  
strongly oxidizing agents, and are  
used in the synthesis of many organic  
compounds. The chloride is also used  
in the synthesis of aluminium chloride.  
The compound is a colorless liquid, boiling at  
100°C. It is soluble in benzene & ether.



latter has been subjected to the Fries-Drafts reaction.

It should be said, the product obtained is the same as that previously obtained.

Twenty grams of the chloride are dissolved in about 200 cubic centimeters of benzene. About 10 grams of aluminum chloride are added in portions at intervals of about two hours. The whole mixture remains about 24 hours in the dark. The flask is heated in a water bath for 24 hours and the mixture is concentrated.

As soon as the benzene becomes yellowish the fluorescence increases. As the fluorescence increases as the action proceeds. The benzoic acid seems to be evolved. The mixture is concentrated consisting of a white solid and a liquid. The solid is a white mass



at the bottom of the flask are poured  
a mixture of benzene and water, with an excess  
of water, funnel <sup>the</sup> separating the benzene  
layer the water, which has become  
milky in appearance, is again extracted  
several times with benzene. The benzene  
is evaporated <sup>2</sup> and the residue is  
dissolved in alcohol, in  
benzene and charcoal decolorizes the  
solution to some extent but the product  
remains colorless and fairly pure. It  
is crystalline and melts at 52  
after several crystallizations, and is  
a white solid obtained in small amount  
from

0.4856 gram of substance



Calculated for

*Found*

Pa. 36

17. 03





of the compound, and the results.

### Qualitative Analysis

In making determinations of carbon  
the hydrogen in these compounds, a  
series of trials were made, and the  
results were always  
found to be higher than the theoretical  
values. These determinations  
showed that a certain amount of  
which is absorbed by the reagent, the  
results are equal to the theoretical  
values. The difficulty in the determination of  
carbon and hydrogen is due to the presence of  
water in the compound. The  
water present in the compound is found  
to be a certain amount, and the  
results are found to be higher than the  
theoretical values. The difficulty in making com-



Solutions of cane sugar. In each case the difficulty is removed by the introduction of a tube containing a solution of silver cyanate in sulphuric acid, in the apparatus for the purification of the oxygen.

Determinations of carbon and nitrogen in the products of the reaction, and in the starting material, are made by the method of Dumas, and in the case of the products, the results are given in the following table. The results of the analysis of the products are not complete after heating for several days at  $310^{\circ}$ , crystals are breaking upon the sides of the tube. This will give some idea of the stability of these compounds.

Solutions of phenylthioacetic acid, phenylthioacetic acid, phenylthioacetic acid, phenylthioacetic acid, phenylthioacetic acid.



acid. These substances described by Dr. L. G. S. & others, are all excellent food material for fungi. In fact, it is in some cases difficult to know what solutions of acids the fungi which develop from the same solution are. In fact, the fungi show little tendency to fruit, showing further that these acids are not the material.



## Conclusions.

1. The compound described by Strahl <sup>and</sup> Schulze is not a sulphone, <sup>as</sup> is not therefore to be represented by the formula
- $$C_6H_4 \begin{matrix} \nearrow COOH \\ \searrow SO_2 C_6H_5 \end{matrix} \cdot H_2O.$$

The grounds for this statement are:—

(a) The compound is capable of <sup>being</sup> oxidation without difficulty.

(b) Although, as at first obtained, it appears to be an individual substance, it can be broken down by simple crystallization from alcohol into two constituents, one of which is a sulphonic acid, the other carbonic acid.

2. When phenylthioacetic acid is treated with dilute nitric acid, the principal product is phenylsulphonic acid & carbonic acid.





4. The acid is obtained by the oxidation of phenylhydrazide with chromic acid or by means of potassium permanganate, a product is obtained which appears to have the formula  $C_{10}H_{10}O_4$ .

It is apparently identical with the acid obtained by the oxidation of phenylhydrazide.

5. This acid yields benzophenone on treatment with concentrated sulphuric acid.

6. By treatment with hydrophorus sulphide a crystalline compound is obtained which also appears to be identical with the compound obtained by the oxidation of phenylhydrazide.

7. By subjecting the chloride to anhydrous  $SO_2$  the acid is obtained which



and were found identical with that obtained by Eimeen & Ganted.

Evidence in favor of the structure of the compound  $C_6H_4 \begin{smallmatrix} C_6H_5O_2H \\ \diagup \end{smallmatrix} SO_2 \begin{smallmatrix} C_6H_5 \\ \diagdown \end{smallmatrix}$  obtained by Eimeen & Ganted.

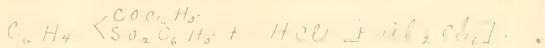
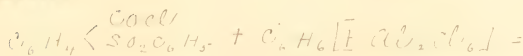
a) Its formation from orthotolylbenzenesulphonic acid:

$C_6H_4 \begin{smallmatrix} C_6H_5 \\ \diagup \end{smallmatrix} SO_2 \begin{smallmatrix} C_6H_5 \\ \diagdown \end{smallmatrix} + H_2O \rightarrow C_6H_4 \begin{smallmatrix} C_6H_5O_2H \\ \diagup \end{smallmatrix} SO_2 \begin{smallmatrix} C_6H_5 \\ \diagdown \end{smallmatrix}$   
 from orthotoluenesulphonyl chloride:



$HCl$  (observed)

b) The transformation of the chloride of the acid into orthobenzoyldichlorobenzene:



The latter compound had been obtained



primarily by Lincomb & Saunders by substituting the symmetrical chloride of acetylene dibenzene as the starting material.

Crafts reaction:



Evidence in favor of the structure of the compound  $C_6H_4 \begin{smallmatrix} \text{COOH} \\ \diagdown \end{smallmatrix} \begin{smallmatrix} \diagup \\ \text{SO}_2 \cdot \text{C}_6\text{H}_5 \end{smallmatrix}$  obtained in this modification:

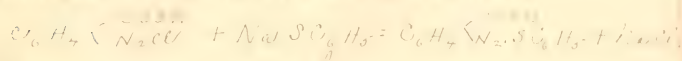
(a) Its method of formation:

(b) Its transformation with methylenedioxybenzoic acid:



By the action of thiocarbonyl in alkaline solution upon this product a new

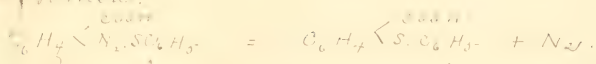
group was formed as follows:



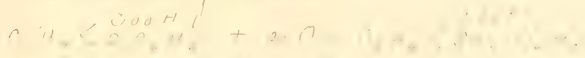
From naming, the diazo compound



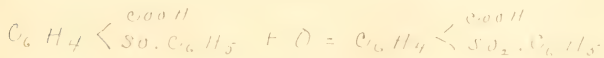
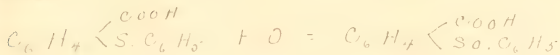
lose nitrogen. Phenylthiocyclohexanoic acid is formed:



On the oxidation of phenylthiocyclohexanoic acid by means of potassium permanganate, benzoyl diphenylthioacetic acid is formed:-



On the action of nitric acid on phenylthiocyclohexanoic acid, benzoyl diphenylthioacetic acid is obtained, which upon further oxidation by means of potassium permanganate, gives benzoyl diphenylthioacetic acid:







- This argument changes the basis of our  
 extent upon the validity of the structure  
 given to phenylthioacetic acid. That  
 the reaction given as a general one for the  
 formation of sulphides was shown by  
 Gracie<sup>(1)</sup> Mann<sup>(2)</sup> and also Stadler<sup>(3)</sup>

Geigler<sup>(4)</sup> utilized it in the preparation  
 of benzophenone. Further, the trans-  
 formation of phenylthioacetic acid  
 into chloral hydrate<sup>(5)</sup> & its related com-  
 pounds, as described on pages 7, 8, & 9,  
 leaves no doubt as to its structure.

(b) Its transformation into benzophenone  
 which is



which had been obtained by Gracie<sup>(1)</sup>  
 Schultze by the oxidation of<sup>(6)</sup> chloral hydrate



(c) Its behavior when fused with

<sup>(1)</sup> Ber. d. Chem. Ges. 15, 1683.

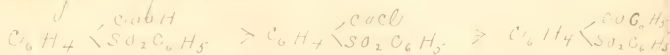
<sup>(2)</sup> Ber. d. Chem. Ges. 15, 1683.

<sup>(3)</sup> Ber. d. Chem. Ges. 23, 2471.



potash: salicylic acid is obtained along with the usual decomposition products of phenylsulphonic acid.

(b) Its transformation into monobenzoyl diphenylsulphonic acid

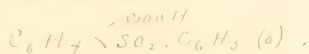


the latter being identified by melting point, analysis, & behavior toward such reagents as ferric chloride, ferric bromide, & benzoyl chloride.

It is not an ester of salicylic acid, as these latter are easily saponified by alkali, & the reaction is reversible. The reaction is irreversible, & the product is a new compound. The same argument holds for the sulphonic compound prepared by Levene & Scott.



The evidence seems convincing that  
the two compounds are to be represented  
by the formula



At present there is no theory which  
accounts for the existence of such  
structures. It is of course possible, by ex-  
tending our conceptions of stereochemistry  
to the case of such structures, to  
account for two enantiomers of these meso-  
structures. Besides, no other cases analogous  
to the above have been observed, and the  
development of such ideas seems unwarranted.  
Besides, in such cases we  
should expect an easy transformation  
from one compound to the other, which  
is not the case here the only possible  
case being that of ortho-hydroxybenzoic  
acids which is obtained from the









identical with the one obtained previously  
by Michael & Rabin<sup>24</sup> & also by Otto.<sup>(17)</sup>

In 1890 Leinen<sup>25</sup> & Nevill<sup>(18)</sup> prepared  
the same sulphone by treating paratoluene-  
sulphonylchloride with benzene<sup>25</sup> & alums-  
in solution.

There are other accounts of the study  
of which would also require to be kept  
in mind. The constants  
characterizing a substance such as its melting  
point would be of interest, as well as  
the subject of the sulphone obtained  
by Leinen & Nevill to the action of  
concentrated sulphuric acid<sup>25</sup> to the  
decomposition by means of caustic pot-  
ash.



## Biography

William Stone Weedon was born  
Salvert County, Maryland, September  
5, 1877. His early education was ob-  
tained in the public schools of Baltimore.  
In 1895 he entered the Maryland  
Agricultural College where he remained  
three years to 1897. In 1898  
In October 1897 he entered the Johns  
Hopkins University as a graduate stu-  
dent in Chemistry. The following year  
he was elected as a member of the  
Sigma Xi Society at the University of  
Maryland. During the summer  
of 1898 he was assistant in the department  
at the Baltimore City College. In  
October 1898 he returned to work at  
the Johns Hopkins University. During



chemist and lecture assistant in the  
Mineralogical Museum. During the same year he  
was instructor in Chemistry in Leich-  
man's College-Preparatory School, Balti-  
more. In 1882 he received a fellowship  
in Chemistry at the University of  
subordinate subjects such as Botany  
and Mineralogy.































